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(54) Title: **CHEWING GUM COMPOSITIONS**

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(57) Abstract: The present invention relates to chewing gum composition comprising a polymeric surface active agent. A second aspect of this invention relates to a crunchy chewing gum wherein the crunchy texture is provided by particulate polyphosphate particles within the formulation and which lasts throughout the initial minutes of mastication. The chewing gum composition may also contain a cationic material and/or an orally active metallic ion. The chewing gum composition will provide surface conditioning effects on a subject's teeth and/or oral mucosa and the "crunchy" texture is used as a sensate to reinforce these effects. The surface conditioning effects can be measured through in vitro or in vivo testing. The in vitro testing shows a total surface energy and/or a lewis base score to increase immediately after treatment with the chewing gum and then decrease over time. The in vivo testing shows a water contact angle of the oral mucosa to decrease after treatment with the chewing gum composition and/or a significantly higher smooth teeth feel relative to other chewing gum compositions. The present invention also relates to methods of providing surface conditioning effects to a subject comprising administering to the subject a chewing gum comprising a polymeric surface active agent. The present invention also relates to methods of reducing astringency of a chewing gum containing an orally active metallic ion without significantly reducing the efficacy of the metallic ion.

## CHEWING GUM COMPOSITIONS

## CROSS-REFERENCE

This application claims priority to Provisional Application Serial No. 60/180,352, filed February 4, 2000 and PCT Application No. PCT/US00/17177, filed June 22, 2000 in English.

## BACKGROUND OF THE INVENTION

The present invention relates to chewing gum compositions containing polymeric surface active agents, including polyphosphates. These polymeric surface active agents may provide novel surface conditioning reaction to oral surfaces such as the teeth and mucosa. This leads to improved cleaning impression. The present invention further relates to compositions wherein the chewing gum has a crunchy texture conferred to it by the polyphosphate particles.

Polymeric surface active agents, such as polyphosphates, are known in the oral care art. Although disclosed in chewing gum compositions, these ingredients are most commonly found in dentifrice compositions. Polyphosphates are known to provide an anti-calculus benefit as stated in U.S. Patent 5,094,844, US 4808401 and EP 0333301 issued to Gaffar et al.. Polyphosphates are also known to provide a buffering effect within zinc comprising oral compositions as stated in U.S. Patent 4,170,632 and US 4,170,633 both to Wagenknecht, deceased et al. Other chewing gum patents disclosing a polyphosphate include U.S. patent 5,702,687 issued to Miskewitz and EP 0,387,024 issued to McClanahan.

Prior art also exists wherein particulate matter has been incorporated into chewing gum compositions such that the resultant product does have a somewhat "crunchy" texture. For example chewing gums with a "crunchy" exterior coating have been previously disclosed such as the hard sugar coated gums disclosed in US 4,486,511 and US 4,792,453 and gums with surface printed solid particles disclosed in US 3,962,463. Such disclosures are limited to a "crunchy" exterior surface of the gum and thus do not provide an overall texture sensation. Furthermore, confectionery gums wherein sugars and sugar substitutes (such as isomalt, candy and the like), sometimes in conjunction with freeze dried food stuffs, are distributed throughout the body of the gum for the purpose, at least in part, of providing a "crunchy" texture are also known (FR 2,748,902; GB 950,811; EP 017,691; US 5,958,472, and US 5,017,385). Whilst these latter disclosures do provide useful advances in conferring a "crunchy" sensation to the gum they do so by the use of food stuffs and not by use of the oral care active itself.

Surprisingly, research has now revealed additional benefits of chewing gums containing polymeric surface active agents, particularly when the polymeric surface active agent is a polyphosphate, even more particularly a particulate polyphosphate. These new benefits are related to effects on the surface chemical characteristics of mucosal and tooth surfaces which provide remarkable cleaning impression and positive mouth feel characteristics for extended

periods of time during and following use. These effects have now been shown to be correlated to effects on oral surface energy characteristics including modification of surface hydrophilic and hydrophobic properties. Additionally, when certain polyphosphates, particularly of a particulate nature, are incorporated within the gum they can confer a "crunchy" texture to the product which lasts throughout the initial minutes of mastication and which reinforces for the consumer the oral care benefit of the product. Furthermore, due to the soluble nature of the polyphosphate materials, the "crunchy" texture disappears over time leaving no gritty residue.

The surface conditioning effects can be measured using several different methods. The surface conditioning effects on a subject's teeth and oral mucosa can be measured in vivo. These measurements include consumer responses on questions concerning clean teeth and smooth teeth. Other in vivo measurements include the water contact angle on the tooth surface and on the mucosa surfaces. The surface conditioning effects can also be measured in considerable detail through in vitro methods. In vitro methods are made over time to measure surface free energies and pellicle film thickness and composition.

It has also been found that the polymeric surface active agent can help to reduce the astringency of a metallic ion. Additionally, this reduction in astringency can occur without significantly reducing the efficacy of the metallic ion and without significantly reducing the efficacy and the surface conditioning effects of the polymeric surface active agent.

It is an object of the present invention to provide chewing gum compositions containing polymeric surface active agents which provide improved intraoral cleaning impression and smooth tooth surface impression derived from the chemical control of tooth and mucosal surface energy characteristics.

It is an object of the present invention to provide chewing gum compositions comprising polymeric surface active agents, cationic material, and/or orally active metallic ions.

It is also an object of the present invention to provide chewing gum compositions with reduced astringency, but wherein the efficacy of the chewing gum is not significantly reduced.

It is a further object of this invention to provide a chewing gum which has a "crunchy" texture during the initial minutes of mastication disappearing over time to leave no gritty residue and wherein this "crunchy" texture is provided by a polyphosphate.

These and other objects of the present invention will become readily apparent from the detailed description which follows.

All percentages and ratios used herein are by weight of the specific chewing gum composition, unless otherwise specified. All measurements are made at 25° C, unless otherwise specified.

#### SUMMARY OF THE INVENTION

A first aspect of this invention relates to a chewing gum composition comprising a polymeric surface active agent. The chewing gum composition may also contain a cationic

material and/or an orally active metallic ion. The chewing gum composition will provide surface conditioning effects on a subject's teeth and/or oral mucosa. The surface conditioning effects can be measured through in vitro or in vivo testing. The in vitro testing shows a total surface energy and/or a lewis base score to increase immediately after treatment with the chewing gum and then decrease over time. The in vivo testing shows a water contact angle of the oral mucosa to decrease after treatment with the chewing gum composition and/or a significantly higher smooth teeth feel relative to other chewing gum compositions. The present invention also relates to methods of providing surface conditioning effects to a subject comprising administering to the subject a chewing gum comprising a polymeric surface active agent. The present invention also relates to methods of reducing astringency of a chewing gum containing a metallic ion without significantly reducing the efficacy of the metallic ion.

According to a second aspect this invention relates to a chewing gum composition which has a crunchy texture wherein the crunchy texture is conferred to the gum by the inclusion of particulate polyphosphate material within the composition. The "crunchy" texture can be attained when the polyphosphate material used has a particle size of between 100 $\mu$ m and 2000 $\mu$ m. Furthermore, by ensuring that the polyphosphate is at least sparingly soluble the "crunch" will slowly disappear to leave a non gritty gum.

#### DETAILED DESCRIPTION OF THE INVENTION

The chewing gum compositions of the present invention may be in the form of a conventional chewing gum or any other product form which is suitable for chewing. Suitable physical forms include sticks, dragees, chiclets, and batons. The chewing gum may also be a digestible or dissolvable gum suitable for chewing. A chewing gum is typically retained in the oral cavity for a time sufficient to allow ingredients released to contact substantially all of the dental surfaces and/or oral tissues for purposes of oral activity.

The term "carrier materials" as used herein means any safe and effective additional chewing gum components used in the chewing gum compositions of the present invention. Such materials include abrasive polishing materials, elastomers, resins, plasticisers, fats, solvents, waxes, emulsifiers, softeners, bulking agents, sweeteners, absorbents, orally active metallic ions, cationic material, fluoride ion sources, additional anticalculus agents, antimicrobial agents, buffers, whitening agents, alkali metal bicarbonate salts, thickening materials, humectants, water, surfactants, titanium dioxide, flavoring agents, xylitol, coloring agents, and mixtures thereof.

The present compositions comprise essential components, as well as optional components. The essential and optional components of the compositions of the present invention are described in the following paragraphs.

#### Polymeric Surface Active Agent

The present invention includes a polymeric surface active agent. The agents will provide the surface conditioning effects. These agents may also be tartar control or anticalculus agents and may also provide stain control and reduction in plaque. The polymeric surface active agents will also provide a clean teeth and longer lasting clean teeth and mouthfeel.

The polymeric surface active agents include any agent which will produce the desired surface conditioning effects. The desired surface conditioning effects include the desorption of adsorbed pellicle proteins and creating a hydrophilic tooth surface immediately after treatment. These surface conditioning effects are measured through in vitro methods over time. The agents will also create several related surface conditioning effects which are measured through in vivo testing done over time. The effects include creating an increased hydrophilic surface on the tooth surface and a hydrophobic surface on the oral mucosa as measured by changed water contact angles. It is thought that these measurements will correlate with consumers noticing a cleaning impression.

The polymeric surface active agents include the phosphorylated polymers, polyelectrolytes, and acrylate polymers. Additional polyelectrolytes include the antibacterial enhancing agents described in U.S. patents 5,032,386 and 5,840,281, both to Gaffar et al. A preferred polymeric agent is polyvinylphosphonic acid. Other suitable polymeric surface active agents include the polymers described in U.S. patents 5,292,501; 5,213,789; 5,093,170; 5,009,882; and 4,939,284; all to Degenhardt et al. and U.S. patent 5,011,913 to Benedict et al. A preferred polymer is diphosphonate modified polyacrylic acid. Polymers with activity must have sufficient surface binding propensity to desorb pellicle proteins and remain affixed to intraoral surfaces. For tooth surfaces, polymers with end or side chain phosphate or phosphonate functions are preferred, although other polymers with mineral binding activity may prove effective depending upon adsorption affinity.

The preferred polymeric surface active agent is a polyphosphate. A polyphosphate is generally understood to consist of two or more phosphate molecules arranged primarily in a linear configuration, although some cyclic derivatives may be present. Although pyrophosphates and tripolyphosphate are a polyphosphates, the polyphosphates desired are those having around four or more phosphate molecules so that one or more internal phosphate groups may be present. The pyrophosphates are discussed separately under anticalculus agents. The inorganic polyphosphate salts desired include tetrapolyphosphate and hexametaphosphate, among others. Polyphosphates larger than tetrapolyphosphate usually occur as amorphous glassy materials. Preferred in this invention are the linear "glassy" polyphosphates having the formula:



wherein X is sodium, potassium, or hydrogen and n averages from about 6 to about 125. For use in the second aspect of this invention it is preferred that the particulate polyphosphate is sodium polyphosphate with an average chain length of from about 10 to about 30, preferably from about

15 to 25, more preferably from about 21 to about 23. Preferred are polyphosphates manufactured by FMC Corporation which are commercially known as Sodaphos ( $n \approx 6$ ), Hexaphos ( $n \approx 13$ ), and Glass H ( $n \approx 21$ ). Hexaphos and Glass H are preferred with Glass H being the most preferred polyphosphate. These polyphosphates may be used alone or in a combination thereof. The phosphate sources are also described in more detail in Kirk & Othmer, *Encyclopedia of Chemical Technology*, Fourth Edition, Volume 18, Wiley-Interscience Publishers (1996), incorporated herein by reference in its entirety, including all references incorporated into Kirk & Othmer.

The amount of polymeric surface agent required is an effective amount to provide the surface conditioning effects and, if desired, according to a second aspect of the invention, to provide a "crunchy" texture. An effective amount of a polymeric surface active agent will typically be from about 0.1% to about 50%, preferably from about 1% to about 35%, more preferably from about 2% to about 25%, and most preferably from about 5% to about 15% by weight of the total chewing gum composition. In addition to creating the surface conditioning effects, the polymeric surface active agent has been found to associate with orally active metallic ions or species whilst maintaining their solubility and efficacy. For example, the polymeric surface active agent may form a complex with stannous fluoride or zinc and still provide the desired tartar control, stain control, and surface conditioning, in addition to not significantly prohibiting the efficacy of the stannous fluoride or zinc. The polymeric surface active agents may enhance the solubility of orally active metallic ions, such as zinc salts and stannous salts. The orally active metallic ions may provide several efficacious benefits to the chewing gum such as reduced gingivitis, plaque, and sensitivity and improved breath. In addition to not significantly reducing the efficacy of an orally active metallic ion, the polymeric surface active agents, particularly sodium polyphosphate, may reduce the amount of astringency created by the orally active metallic ion. This may be due to lower levels of mucosal protein precipitation in the mouth. This can be measured by in vivo testing and sensory scores. The preferred orally active metallic ions are zinc and tin or stannous. The orally active metallic ions are typically present in an amount of from about 0.01% to about 10%, preferably from about 0.05% to about 5%, more preferably from about 0.1% to about 1%, by weight of the chewing gum.

It may be desirable to have a sustained release of the polymeric surface active agent from the chewing gum. This may be accomplished by incorporating a cationic material whose polymeric surface active agent salt is less soluble in water than the sodium, potassium or hydrogen salts. By adding such cationic material, particularly divalent cationic materials such as calcium, the release rate of the polymeric surface active agent may be tailored to a required profile. The maximum level of cationic material incorporated is one cation per monomer unit forming the polymeric surface active agent. The level of cationic material incorporated is more preferably less than 0.5 cations per monomer unit forming the polymeric surface active agent.

By weight percent, the polymeric surface active agent is generally present in at least about two times the cationic material, preferably about four times the cationic material, and more preferably at least about five times the cationic material. For example, the cationic material is typically present in an amount of up to about 10%, preferably from about 0.05% to about 5%, and more preferably from about 0.1% to about 3%, by weight of the chewing gum.

The particulate polyphosphate material preferred for use in the second aspect of the present invention should have a minimum particle size such that they are retained by a 0.1mm mesh, preferably a 0.112mm mesh, more preferably a 0.16mm mesh, even more preferably a 0.18mm mesh and most preferably a 0.2mm mesh wherein the meshes are selected from the DIN 4188 mesh series. Furthermore the solid particulate materials for use in the second aspect of the present invention should have a maximum particle size such that pass through a 2mm mesh, preferably a 1mm mesh, more preferably an 0.8mm mesh, even more preferably a 0.5mm mesh and most preferably a 0.4mm mesh, again wherein the meshes are selected from the DIN 4188 mesh series.

The solubility of the particulate polyphosphate preferred for use in the second aspect of the present invention should be at least 5g per 100ml at 25°C, preferably of at least 8g, more preferably at least 10g, even preferably at least 15g per 100ml at 25°C. Thus the solid particulate should be "sparingly soluble", or preferably more soluble, wherein the term is defined as in the British Pharmacopoeia, 1999, Volume 1. Whilst there is no limit on the upper solubility of the polyphosphate it is preferred that it is not freely soluble in water otherwise it will dissolve too rapidly for a crunch to be experienced.

It is preferred that particulate polyphosphate materials for use in the second aspect of the present invention have hardness of greater than 1, preferably of 2 or greater, on the Mohs hardness scale. It is also preferred that the particulate polyphosphate materials for use in the second aspect of this invention are distributed evenly throughout the gum base. Additionally it is also preferred that, according to the second aspect of this invention, that the weight ratio of gum to particulate polyphosphate is in the range from about 10:1 to about 1:10, preferably from about 5:1 to about 1:5, more preferably from about 5:1 to about 1:1.

#### Additional chewing gum components

In preparing the present chewing gum compositions, it is desirable to add one or more additional chewing gum components. Such materials are well known in the art and are readily chosen by one skilled in the art based on the physical and aesthetic properties desired for the chewing gum compositions being prepared. These carriers may be included at levels which do not interfere or prohibit the surface conditioning. The amount of polymeric surface active agent may be increased to account for the additional components. The additional chewing gum components typically comprise from about 30% to about 99%, preferably from about 40% to

about 98%, and more preferably from about 70% to about 95%, by weight of the chewing gum composition.

Additional chewing gum components include carrier materials. The carrier materials are water insoluble materials which are typically not released in the mouth and water soluble materials which are released in the mouth. Water insoluble materials are typically used to form a chewing gum base.

An abrasive polishing material may be included in the chewing gum compositions. The abrasive polishing material contemplated for use in the compositions of the present invention can be any material which does not excessively abrade dentin. The abrasive polishing material should be formulated in the chewing gum composition so that it does not compromise the stability of any ingredients. Typical abrasive polishing materials include silica gels and precipitates; aluminas; water soluble phosphates (including orthophosphates, polymetaphosphates, and pyrophosphates); and mixtures thereof. Specific examples include dicalcium orthophosphate dihydrate, calcium pyrophosphate, tricalcium phosphate, calcium polymetaphosphate, insoluble sodium polymetaphosphate, hydrated alumina, beta calcium pyrophosphate, calcium carbonate, and resinous abrasive materials such as particulate condensation products of urea and formaldehyde. Mixtures of abrasives may also be used. The abrasive in the chewing gum compositions is generally from about 1% to about 70% and preferably from about 5% to about 50%, by weight of the chewing gum composition.

Another ingredient of the chewing gum composition is an elastomer or elastomer mixture. The elastomers useful in the present composition include styrene-butadiene rubber (SBR) and other elastomeric materials generally known in the art. Illustrative elastomers include SBR, synthetic gums or elastomers such as polyisobutylene and isobutylene-isoprene copolymers; natural gums or elastomers such as chicle, natural rubber, jelutong, balata, guttapercha, lechi caspi, sorva and mixtures thereof. The elastomer or elastomer mixture is generally present in an amount of from 2% to about 30% and preferably from about 5% to about 25% by weight. When the total amount of elastomer is below 2% the base composition lacks elasticity, chewing texture, and cohesiveness whereas at amounts above about 30% the formulation is hard, rubbery and maintains a tight chew.

An optional but desirable ingredient of the chewing gum composition is a resin. The resin serves to plasticise the gum base. Suitable resins for use herein include polyvinyl acetate (PVA) and terpene resins, including polyterpene and polymers of alpha-pinene or beta-pinene, and mixtures thereof. The resin can conveniently be used at a level of from about 3% to about 25%, preferably from about 5% to about 20% by weight of the gum composition.

In addition to the resin component, the gum base compositions of the present invention preferably comprise a plasticiser in an amount up to about 10%, preferably from about 0.1% to



about 3% by weight of the gum composition. Suitable plasticisers include glyceryl triacetate, acetylated monoglyceride, glyceryl tributyrate, ethyl laurate, ethyl acetoacetate, diethyl tartrate, ethyl or butyl lactates, diethyl malate, ethyl oleate, castor oil, succinylated monoglycerides or mixtures thereof. Glyceryl triacetate and acetylated monoglyceride are preferred.

Various fats can also be included in the chewing gum compositions of the present invention. Preferred fats include the hydrogenated vegetable oils such as hydrogenated palm oil, hydrogenated soybean oil, hydrogenated cotton seed oil and various other hydrogenated vegetable oils and mixtures thereof. The fats can suitably be used at a level up to about 20%, preferably from about 1% to about 10% by weight of the chewing gum composition.

A further desirable ingredient of the chewing gum base composition is an elastomer solvent. The elastomer solvent aids in softening the elastomer component. Such elastomer solvents include methyl, glycerol or pentaerythritol esters of rosins or modified rosins, such as hydrogenated, dimerized or polymerised rosins or mixtures thereof. Examples of elastomer solvents suitable for use herein include the pentaerythritol ester of partially hydrogenated wood rosin, pentaerythritol ester of wood rosin, glycerol ester of partially dimerized rosin, glycerol ester of polymerised rosin, glycerol ester of tall oil, wood or gum rosin, glycerol ester of partially hydrogenated rosin, methyl ester of partially hydrogenated rosin, and mixtures thereof. The elastomer solvent can be employed in an amount ranging from about 2% to about 50%, preferably from about 10% to about 35% by weight of the chewing gum.

The gum base compositions can also include one or more waxes. Suitable waxes include paraffin wax; microcrystalline wax; Fischer-Tropsch paraffin; natural waxes such as candellilla, carnauba and beeswax; polyolefin waxes such as polyethylene wax; and mixtures thereof. The waxes can be present in levels up to about 25%, preferably from about 5% to about 20% by weight of the gum composition.

The chewing gum also preferably includes an emulsifier. Suitable emulsifiers include glycerol monostearate, lecithin, fatty acid monoglycerides, diglycerides, propylene glycol monostearate and mixtures thereof. The emulsifier is employed in amounts up to about 10% and preferably from about 2% to about 6% by weight of the chewing gum.

A variety of softeners can also be employed in the chewing gum compositions of the present invention. Suitable softeners include fatty materials such as lanolin, stearic acid, sodium stearate and potassium stearate; polyhydric alcohols such as glycerine, propylene glycol, and the like; and mixtures thereof. The softeners can suitably be used at a total level of up to about 30%, preferably from about 0.1% to about 10% by weight of the chewing gum. In a preferred embodiment, the chewing gum composition comprises a fatty softener selected from stearic acid, sodium stearate, potassium stearate and mixtures thereof in an amount of from about 0.1% to

about 10% by weight of the chewing gum. Preferably, the fatty softener is stearic acid. The gum base composition may further comprise a polyhydric alcohol. If present, the polyhydric alcohol is present in an amount of from about 0.5% to about 25%, more preferably from about 1% to about 10% by weight of the chewing gum. Such materials, when incorporated into the gum base, assist in modifying the texture and consistency properties. In particular, they help to soften the chew and to maintain chew softness over an extended period of time.

Bulking agents, such as fillers, can also be employed in the chewing gum. Suitable fillers and bulking agents are generally non-abrasive, preferably with an average particle size less than 5  $\mu\text{m}$ , more preferably less than 3  $\mu\text{m}$  and especially less than 1  $\mu\text{m}$ . Illustrative bulking agents include calcium carbonate or ground limestone, talc, aluminium hydroxide, alumina, aluminium silicates, dicalcium phosphate and mixtures thereof. Where present, the filler can be used in levels up to about 50%, preferably up to about 30%, most preferably from up to about 10% by weight of the chewing gum.

Suitable bulk sweeteners are monosaccharides, disaccharides, and polysaccharides such as xylose, ribose, glucose, mannose, galactose, fructose, dextrose, sucrose, sugar maltose, fructo oligo saccharide syrups, partially hydrolysed starch, or corn syrup solids. Preferred sweetening agents are sugar alcohols such as sorbitol, xylitol, mannitol, maltitol, isomalt, hydrogenated starch hydrolysate, inulin, and other non-cariogenic edible polyols such as glycerin and erythritol and mixtures thereof.

In general, the amount of sweetener will vary with the sweetener used and desired amount of sweetener selected for a particular chewing gum. This amount will normally vary from about 0.01% when using a high intensity sweetener to about 80% by weight of the chewing gum composition when using an easily extractable bulk sweetener. The bulk sweeteners described above, are preferably used in amounts of about 10% to about 80% by weight and most preferably about 30% to about 70% by weight. These amounts are ordinarily necessary to achieve a desired level of sweetness independent from the flavor level achieved from the flavoring agents.

In preferred embodiments, the chewing gum composition further comprises a high intensity sweetener. Suitable high intensity sweeteners include: dipeptide based sweeteners such as L-aspartyl-L-phenylalanine methyl ester (Aspartame) and equivalents described in U.S. Pat. No. 3,492,131, L- $\alpha$ -aspartyl-N-(2,2,4,4-tetramethyl-3-thietanyl)-D-alaninamide hydrate (Alitame) and the like; the soluble saccharin salts, i.e., sodium or calcium saccharin salts; cyclamate salts, acesulfame-K and the like; the free acid form of saccharin; chlorinated derivatives of sucrose such as chlorodeoxysucrose and the like; and protein based sweeteners, such as Thaumatin (talin). The high intensity sweeteners described can be added in amounts of from about 0.01% to about 2.0% and most preferably from about 0.05% to about 0.5% by weight

of the chewing gum composition. Using a high intensity sweetener within the gum base may prolong the flavor of the finished gum composition during chewing.

The chewing gum can also include colorants and pigments, such as titanium dioxide. In general, the gum can contain up to about 2% of pigment and / or colorant. Anti-oxidants can also be included in the gum, at a level of up to about 0.5%. Suitable anti-oxidants are butylated hydroxyanisole, butylated hydroxytoluene, propyl gallate, ascorbic acid and tocopherols.

Flavoring agents well known in the chewing gum art can be added to the chewing gum compositions of the invention. These flavoring agents can be chosen from synthetic flavoring liquid and/or oils derived from plants leaves, flowers, fruits and so forth, and combinations thereof. Representative flavoring liquids include: spearmint oil, cinnamon oil, oil of wintergreen (methylsalicylate) and peppermint oils. Also useful are artificial, natural or synthetic fruit flavors such as citrus oil including lemon, orange, banana, grape, lime, apricot and grapefruit and fruit essences including apple, strawberry, cherry, orange, pineapple and so forth; bean and nut derived flavors such as coffee, cocoa, cola, peanut, almond and so forth. Additionally, flavor adsorbed onto a hydrophilic matrix may be included e.g. "spray-dried" flavors. Furthermore encapsulated flavors may be included. Also included in the term flavorant are sensates and coolants. Preferred coolants include MGA, Physcool, WS-3, WS-23, TK-10, and combinations thereof.

The amount of flavorant employed is normally a matter of preference subject to such factors as flavor type, base type and strength desired. In general, amounts up to about 4% by weight and preferably about 0.05% to about 3.0% by weight of the chewing gum composition are usable with amounts of about 0.8% to about 2.5% being preferred.

Water employed in the preparation of commercially suitable chewing gum compositions should preferably be of low ion content and free of organic impurities. Water will generally comprise less than about 10%, preferably from about 0.01% to about 5%, and more preferably from about 0.1% to about 3%, by weight of the composition herein. The amounts of water include the free water which is added plus that which is introduced with other materials, such as with sorbitol, silica, and solutions.

The present invention may also include other agents, such as antimicrobial agents. The chewing gum composition may include an orally active metallic ion as an antimicrobial agent, particularly salts of zinc, tin and silver and copper.

Other antimicrobial agents include the water insoluble non-cationic antimicrobial agents such as halogenated diphenyl ethers, phenolic compounds including phenol and its homologs, mono and poly-alkyl and aromatic halophenols, resorcinol and its derivatives, bisphenolic compounds and halogenated salicylanilides, benzoic esters, and halogenated carbanilides. The water soluble antimicrobials include quaternary ammonium salts and bis-biquanide salts, among

others. Triclosan monophosphate is an additional water soluble antimicrobial agent. The quaternary ammonium agents include those in which one or two of the substitutes on the quaternary nitrogen has a carbon chain length (typically alkyl group) from about 8 to about 20, typically from about 10 to about 18 carbon atoms while the remaining substitutes (typically alkyl or benzyl group) have a lower number of carbon atoms, such as from about 1 to about 7 carbon atoms, typically methyl or ethyl groups. Dodecyl trimethyl ammonium bromide, tetradecylpyridinium chloride, domiphen bromide, N-tetradecyl-4-ethyl pyridinium chloride, dodecyl dimethyl (2-phenoxyethyl) ammonium bromide, benzyl dimethylstearyl ammonium chloride, cetyl pyridinium chloride, cetyl pyridinium saccharinate, quaternized 5-amino-1,3-bis(2-ethyl-hexyl)-5-methyl hexa hydropyrimidine, benzalkonium chloride, benzethonium chloride and methyl benzethonium chloride are exemplary of typical quaternary ammonium antibacterial agents. Other compounds are bis[4-(R-amino)-1-pyridinium] alkanes as disclosed in U.S. Patent 4,206,215, Issued June 3, 1980, to Bailey, incorporated herein by reference. Other antimicrobials such as copper bisglycinate, copper glysinate, zinc citrate, zinc citrate-maleate, zinc lactate, hexetidine, hexamidine, furanones, and phalimido-peroxycaproic acid may also be included. Also useful are enzymes, including endoglycosidase, papain, dextranase, mutanase, and mixtures thereof. Such agents are disclosed in U.S. Patent 2,946,725, Jul. 26, 1960, to Norris et al. and in U.S. Patent 4,051,234, September. 27, 1977 to Gieske et al., incorporated herein by reference. Specific antimicrobial agents include chlorhexidine, triclosan and its derivatives including triclosan monophosphate, triclosan diphosphate, and phenolated triclosan and flavor oils such as thymol, geraniol, eugenol, and biosol. Triclosan and other agents of this type are disclosed in Parran, Jr. et al., U.S. Patent 5,015,466, issued May 14, 1991, and U.S. Patent 4,894,220, Jan. 16, 1990 to Nabi et al., incorporated herein by reference. These agents may be present at levels of from about 0.01% to about 10%, preferably from about 0.05% to about 5%, and more preferably from about 0.1% to about 2%, by weight of the chewing gum composition.

Optional agents that may be used in combination with the polymeric surface active agent include materials known to be effective in reducing calcium phosphate mineral deposition related to calculus formation. Pyrophosphate salts may be used in the present invention as anticalculus agents or as buffering agents, as long of the surface conditioning effects of the polymeric surface active agent is not eliminated. The amount of pyrophosphate salt useful in making these compositions is any tartar control effective amount, and is generally from about 1.5% to about 15%, preferably from about 2% to about 10%, and most preferably from about 2.5% to about 8%, by weight of the chewing gum composition. Other agents included are synthetic anionic polymers [including polyacrylates and copolymers of maleic anhydride or acid and methyl vinyl ether (e.g., Gantrez), as described, for example, in U.S. Patent 4,627,977, to Gaffar et al., the disclosure of which is incorporated herein by reference in its entirety; as well as, e.g., polyamino propoane sulfonic acid (AMPS)], zinc

citrate trihydrate, diphosphonates (e.g., EHDP; AHP), polypeptides (such as polyaspartic and polyglutamic acids), and mixtures thereof.

The present invention may also include buffering agents to adjust the pH of the chewing gum and may help to stabilize the polymeric surface active agent. Other potential ingredients include a fluoride ion source. An alkali metal bicarbonate salt, surfactants, whitening agents such as peroxide or percarbonate, coloring agents, xylitol, thickening materials, binders, humectants, absorbents such as activated carbon, silica absorbents, cyclodextrins, and zeolites and combinations thereof, may also be included in the chewing gum composition.

The chewing gum composition may be in the form of a chiclet or other form that contains a outer coating or shell around the central portion or gum base of the chewing gum. The outer coating may be hard or crunchy. Typically, the outer coating will be comprised of about sorbitol, malitol, xylitol, isomalt, and other crystallisable polyols. The outer coating may also contain small amounts of water and gum arabic. A polyol coating can be further coated with wax. The present invention may have the polymeric surface active agent present in the chewing gum base, the outer coating, or both.

It may be desirable to have a rapid release of the polymeric surface active agent from the chewing gum. This may be accomplished by incorporating some or all of the polymeric surface active agent into the outer coating. To further increase the rate of release of the polymeric surface active agent from the outer coating, the particles are processed in a manner to result in a microporous sized porosity.

#### Method of Treatment

The present invention relates to methods for providing surface conditioning effects. The present invention also relates to methods of providing clean mouth and tooth feel, smooth teeth feel, and longer lasting clean or smooth tooth feel. Additional efficacy, such as reduced gingivitis, plaque, calculus, and sensitivity, and improved breath may also occur depending upon the composition of the chewing gum. The benefits of these compositions may increase over time when the composition is repeatedly used.

The method of treatment herein comprises contacting a subject's dental enamel surfaces and mucosa in the mouth with the chewing gum compositions according to the present invention. The method of treatment will typically be by chewing the gum. The subject may be any person or lower animal whose tooth surface contact the chewing gum composition.

#### Examples & Method of Manufacturing

The following examples further describe and demonstrate embodiments within the scope of the present invention. These examples are given solely for the purpose of illustration and are not to be construed as limitations of the present invention as many variations thereof are possible without departing from the spirit and scope.

Material Name (INCI)	A	B	C Core	Coating	D Core	Coating
Sorbitol	25.850	18.350	33.350		33.350	
Xylitol	16.700	16.700	16.700		16.700	
Gum base (e.g. Prestige-PL, Cafosa)	28.000	28.000	28.000		28.000	
Sodium polyphosphate, n=21; Glass H (FMC)	7.500	15.000	0.000	37.500	0.000	
Glass H (microporous sized)						37.500
Hydrogenated starch hydrolysate (85% solids)	8.000	8.000	8.000		8.000	
Glycerin	7.000	7.000	7.000		7.000	
Mannitol	5.000	5.000	5.000		5.000	
Maltitol				62.250		62.250
Ethyl cellulose (Ethocell, Dow Corning)						
Zinc lactate dihydrate						
Sodium polyphosphate, n=6; Sodaphos (FMC)						
Sodium polyphosphate, n=13; Hexaphos (FMC)						
Poloxamer 407						
Flavor	1.600	1.600	1.600	0.250	1.600	0.250
Aspartame	0.200	0.200	0.200		0.200	
Spray dried flavor	0.150	0.150	0.150		0.150	
<b>TOTAL</b>	<b>100.000</b>	<b>100.000</b>	<b>100.000</b>	<b>100.000</b>	<b>100.000</b>	<b>100.000</b>
	0	0		0		
<b>% OF TOTAL COMPOSITION</b>	<b>100.000</b>	<b>100.000</b>	<b>80.000</b>	<b>20.000</b>	<b>80.000</b>	<b>20.000</b>
	0	0				

Material Name (INCI)	E	F	G	H	I	J	K
Sorbitol	15.850	25.123	18.350	23.350	23.850	23.850	24.719
Xylitol	16.700	16.700	16.700	16.700	16.700	16.700	16.700
Gum base (e.g. Prestige-PL, Cafosa)	28.000	28.000	28.000	28.000	28.000	28.000	28.000
Sodium polyphosphate, n=21; Glass H (FMC)	7.500	7.500			7.500	7.500	7.500
Hydrogenated starch hydrolysate (85% solids)	8.000	8.000	8.000	8.000	8.000		8.000
Glycerin	7.000	7.000	7.000	7.000	7.000	7.000	7.000
Mannitol	5.000	5.000	5.000	5.000	5.000	5.000	5.000
Maltitol						8.000	
Ethyl cellulose (Ethocell, Dow Corning)	10.000						
Zinc lactate dihydrate		0.727					
Sodium polyphosphate, n=6; Sodaphos (FMC)			15.000				
Sodium polyphosphate, n=13; Hexaphos (FMC)				10.000			
Poloxamer 407					2.000	2.000	

Flavor	1.600	1.600	1.600	1.600	1.600	1.600	1.600
Aspartame	0.200	0.200	0.200	0.200	0.200	0.200	0.200
Spray dried flavor	0.150	0.150	0.150	0.150	0.150	0.150	0.150
Calcium chloride							0.754
<b>TOTAL</b>	<b>100.000</b>	<b>100.00</b>	<b>100.00</b>	<b>100.00</b>	<b>100.00</b>	<b>100.00</b>	<b>100.00</b>
				0	0	0	0
<b>% OF TOTAL COMPOSITION</b>	<b>100.000</b>	<b>100.00</b>	<b>100.00</b>	<b>100.00</b>	<b>100.00</b>	<b>100.00</b>	<b>100.00</b>
				0	0	0	0

### Making Instructions

#### Products A, B

Heat gum base to ~45°C to soften. Maintain mixer vessel cavity at ~45°C during entire mixing process. Add gum base to mixing cavity of double sigma blade mixer and mix for 5 minutes. Add mannitol and spray-dried menthol. Mix for 2 minutes. Add glycerin and mix for 2 minutes. Add 50% of xylitol and mix for 2 minutes. Add hydrogenated starch hydrolysate and mix for 5 minutes. Add 50% sorbitol and mix for 3 minutes. Add second 50% of xylitol, Glass-H and aspartame and mix for 3 minutes. Add flavor and mix for 3 minutes.

#### Products C, D

Heat gum base to ~45°C to soften. Maintain mixer vessel cavity at ~45°C during entire mixing process. Add gum base to mixing cavity of double sigma blade mixer and mix for 5 minutes. Add mannitol and spray-dried menthol. Mix for 2 minutes. Add glycerin and mix for 2 minutes. Add 50% of xylitol and mix for 2 minutes. Add hydrogenated starch hydrolysate and mix for 5 minutes. Add 50% sorbitol and mix for 3 minutes. Add second 50% of xylitol aspartame and mix for 3 minutes. Add flavor and mix for 3 minutes. Store for ~1 week at 15-20°C, 30-60% RH (conditioning). Tumble chewing gums in coater-drier with maltitol solution and Glass-H. Maintain tumbling until surface is dry.

#### Product E

Heat gum base to ~45°C to soften. Maintain mixer vessel cavity at ~45°C during entire mixing process. Add gum base to mixing cavity of double sigma blade mixer and mix for 5 minutes. Add mannitol and spray-dried menthol. Mix for 2 minutes. Add glycerin and mix for 2 minutes. Add 50% of xylitol and mix for 2 minutes. Add hydrogenated starch hydrolysate and mix for 5 minutes. Add 50% sorbitol and mix for 3 minutes. Add second 50% of xylitol, Glass-H in encapsulated ethyl cellulose and aspartame and mix for 3 minutes. Add flavor and mix for 3 minutes.

#### Product F

Heat gum base to ~45°C to soften. Maintain mixer vessel cavity at ~45°C during entire mixing process. Add gum base to mixing cavity of double sigma blade mixer and mix for 5 minutes.

Add mannitol and spray-dried menthol. Mix for 2 minutes. Add glycerin and mix for 2 minutes. Add 50% of xylitol and mix for 2 minutes. Add hydrogenated starch hydrolysate and mix for 5 minutes. Add 50% sorbitol and mix for 3 minutes. Add second 50% of xylitol, Glass-H, zinc lactate, and aspartame and mix for 3 minutes. Add flavor and mix for 3 minutes.

Product G, H

Heat gum base to ~45°C to soften. Maintain mixer vessel cavity at ~45°C during entire mixing process. Add gum base to mixing cavity of double sigma blade mixer and mix for 5 minutes. Add mannitol and spray-dried menthol. Mix for 2 minutes. Add glycerin and mix for 2 minutes. Add 50% of xylitol and mix for 2 minutes. Add hydrogenated starch hydrolysate and mix for 5 minutes. Add 50% sorbitol and mix for 3 minutes. Add second 50% of xylitol, sodaphos, hexaphos, and aspartame and mix for 3 minutes. Add flavor and mix for 3 minutes.

Product I

Heat gum base to ~45°C to soften. Maintain mixer vessel cavity at ~45°C during entire mixing process. Add gum base to mixing cavity of double sigma blade mixer and mix for 5 minutes. Add mannitol and spray-dried menthol. Mix for 2 minutes. Add glycerin and mix for 2 minutes. Add 50% of xylitol and mix for 2 minutes. Add hydrogenated starch hydrolysate and mix for 5 minutes. Add 50% sorbitol and mix for 3 minutes. Add second 50% of xylitol, Glass-H, Poloxamer 407, and aspartame and mix for 3 minutes. Add flavor and mix for 3 minutes.

Product J

Heat gum base to ~45°C to soften. Maintain mixer vessel cavity at ~45°C during entire mixing process. Add gum base to mixing cavity of double sigma blade mixer and mix for 5 minutes. Add mannitol and spray-dried menthol. Mix for 2 minutes. Add glycerin and mix for 2 minutes. Add 50% of xylitol and mix for 7 minutes. Add 50% sorbitol and mix for 3 minutes. Add second 50% of xylitol, Glass-H and aspartame and mix for 3 minutes. Add flavor and mix for 3 minutes.

Product K

Heat gum base to ~45°C to soften. Maintain mixer vessel cavity at ~45°C during entire mixing process. Add gum base to mixing cavity of double sigma blade mixer and mix for 5 minutes. Add mannitol and spray-dried menthol. Mix for 2 minutes. Add glycerin and mix for 2 minutes. Add 50% of xylitol and mix for 2 minutes. Add hydrogenated starch hydrolysate and mix for 5 minutes. Add 50% sorbitol, calcium chloride and mix for 3 minutes. Add second 50% of xylitol, Glass-H and aspartame and mix for 3 minutes. Add flavor and mix for 3 minutes.



## CLAIMS:

1. A chewing gum composition comprising a polymeric surface active agent wherein the chewing gum provides surface conditioning effects on a subject's teeth which is measured by in vitro testing showing total surface energy to increase immediately after treatment with the chewing gum and then decrease over time.
2. A chewing gum composition comprising a polymeric surface active agent wherein the chewing gum provides surface conditioning effects on a subject's teeth which is measured by in vitro testing showing lewis base score to increase immediately after treatment with the chewing gum and then decrease over time.
3. A chewing gum composition comprising a polymeric surface active agent wherein the chewing gum provides surface conditioning effects on a subject's oral mucosa which is measured by in vivo testing showing a water contact angle to decrease after treatment with the chewing gum.
4. A chewing gum composition comprising a polymeric surface active agent wherein the chewing gum provides surface conditioning effects on a subject's teeth which is measured by in vivo testing showing a significantly higher smooth teeth feel relative to other chewing gums.
5. A method of providing surface conditioning effects to a subject comprising administering to the subject a chewing gum composition comprising a polymeric surface active agent.
6. A chewing gum composition comprising:
  - a. from about 0.1% to about 50% of a polymeric surface active agent;
  - b. up to about 10% of water; and
  - c. from about 40% to about 99% of carrier materials.
7. The chewing gum composition according to Claim 6 wherein the chewing gum has an outer coating.
8. The chewing gum composition according to Claim 6 where the chewing gum further comprises a cationic material.

9. The chewing gum composition according to Claim 8 wherein the cationic material controls the release rate of the polymeric surface active agent.
10. The chewing gum composition according to Claim 8 wherein the cationic material is calcium.
11. The chewing gum composition according to Claim 6 wherein the polymeric surface active agent is a glassy polyphosphate.
12. The chewing gum composition according to Claim 11 wherein the glassy polyphosphate is Glass H.
13. The chewing gum composition according to Claim 6 wherein the chewing gum further comprises an orally active metallic ion.
14. The chewing gum composition according to Claim 13 wherein the astringency of the chewing is reduced by the polymeric surface active agent.
15. The chewing gum composition according to Claim 14 wherein the efficacy of the orally active metallic ion is not significantly reduced by the polymeric surface active agent.
16. A method of reducing astringency of a chewing gum composition containing an orally active metallic ion comprising administering to the subject the chewing gum comprising:
  - a. from about 0.1% to about 50% of a polymeric surface active agent;
  - b. from about 0.01% to about 10% of an orally active metallic ion;
  - c. up to about 10% of water; and
  - d. from about 30% to about 99% of carrier materials;

wherein the efficacy of the orally active metallic ion is not significantly reduced by the polymeric surface active agent.

17. A chewing gum comprising:

(a) from about 0.1% to about 50% of a particulate polyphosphate wherein the particulate polyphosphate:

(i) has a particle size such that it passes through a 2mm mesh and is retained by a 0.1mm;

- (ii) has an aqueous solubility of at least 5g per 100ml at 25°C;
- (b) greater than about 10% gum base comprising one or more elastomers, resins or waxes and mixtures thereof.
18. A composition according to Claim 17 which comprises from about 0.5% to about 30%, preferably from about 1% to about 15%, more preferably from about 5% to about 12%, by weight, of particulate polyphosphate.
19. A composition according to any of Claims 17 to 18 wherein the particulate polyphosphate has a particle size such that it passes through a 1mm mesh, preferably a 0.8mm mesh, more preferably 0.5mm mesh and even more preferably a 0.4mm mesh.
20. A composition according to any of Claims 17 to 19 wherein the particulate polyphosphate has a particle size such that it is retained by a 0.112mm mesh, preferably a 0.16mm mesh, more preferably a 0.18mm mesh and even more preferably a 0.2mm mesh.
21. A composition according to any of Claims 17 to 20 wherein the particulate polyphosphate is sodium polyphosphate with an average chain length of from about 10 to about 30, preferably from about 15 to 25, more preferably from about 21 to about 23.
22. A composition according to any of Claims 17 to 21 wherein the particulate polyphosphate has an aqueous solubility of at least 8g, preferably at least 10g, more preferably at least 15g per 100ml at 25°C.
23. A composition according to any of Claims 17 to 22 wherein the particulate polyphosphate has a hardness of greater than 1, preferably 2, or greater, when measured using the Mohs hardness scale.
24. A composition according to any of Claims 17 to 23 wherein the particulate polyphosphate is dispersed throughout the chewing gum composition.
25. A composition according to any of Claims 17 to 24 wherein the weight ratio of gum to particulate polyphosphate is in the range from about 10:1 to about 1:10, preferably from about 5:1 to about 1:5, more preferably from about 5:1 to about 1:1.

## INTERNATIONAL SEARCH REPORT

Inten Application No

PCT/US 01/03488

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 7 A23G3/30 A61K7/00

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A23G A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, PAJ, WPI Data

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 765 984 A (VELLEKOOP LINDA J ET AL) 23 August 1988 (1988-08-23)  column 1, line 61 -column 2, line 32 column 5, line 55 -column 7, line 49; claims 1,7,8; examples 1,2	1-6, 8-11, 13-19, 24,25
X	EP 0 333 301 A (COLGATE PALMOLIVE CO) 20 September 1989 (1989-09-20) cited in the application page 5, line 43 -page 6, line 7; claim 1; examples 1,5,6 page 4, line 56 - line 58 page 2, line 21 - line 23; claims 1,13  -/--	1-6, 8-11,17

☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

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## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 833 954 A (CHOW LAURENCE C ET AL) 10 November 1998 (1998-11-10)  column 4, line 27 - line 46; claims 1-10 column 8, line 40 - line 53 column 9, line 18 - line 39 column 9, line 63 - column 10, line 11 ---	1-6, 8-10, 13-19, 24
X	US 5 380 530 A (HILL IRA D) 10 January 1995 (1995-01-10) the whole document ---	1-4, 7
X	PATENT ABSTRACTS OF JAPAN vol. 1997, no. 03, 31 March 1997 (1997-03-31) & JP 08 301742 A (EZAKI GLICO CO LTD), 19 November 1996 (1996-11-19) abstract ---	1-4
X	WO 95 07683 A (PROCTER & GAMBLE) 23 March 1995 (1995-03-23) page 10, line 19 - page 11, line 22; examples ---	1-6, 8-10
X	WO 97 02011 A (PROCTER & GAMBLE ; EDWARDS MARK IEUAN (GB); HUGHES IAIN ALLAN (GB)) 23 January 1997 (1997-01-23) the whole document ---	1
A	US 5 407 661 A (SIMONE ALEXANDER J ET AL) 18 April 1995 (1995-04-18) ---	
A	EP 0 414 932 A (CSEMEGE EDESIPARI GYAR) 6 March 1991 (1991-03-06) ---	
A	EP 0 309 414 A (AVANTGARDE SPA) 29 March 1989 (1989-03-29) page 2, line 28 - page 3, line 50 ---	1
A	US 3 932 603 A (HAAS GERHARD J) 13 January 1976 (1976-01-13) column 1, line 19 - line 27; example; tables ---	1
A	US 5 783 172 A (ASCIONE JEAN-MARC ET AL) 21 July 1998 (1998-07-21) column 1, line 25 - column 3, line 58; claims 1, 7, 11, 12; examples ---	1
A	US 4 867 989 A (SILVA JOSE N ET AL) 19 September 1989 (1989-09-19) -----	

## INTERNATIONAL SEARCH REPORT

Information on patent family members

Inten      if Application No

PCT/US 01/03488

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 4765984      A	23-08-1988	US 4705680 A	10-11-1987
		AT 389812 B	12-02-1990
		AT 9487 A	15-07-1989
		AU 598220 B	21-06-1990
		AU 6712887 A	23-07-1987
		BE 1000488 A	27-12-1988
		CA 1286993 A	30-07-1991
		CH 676082 A	14-12-1990
		DE 3701122 A	23-07-1987
		DK 35287 A	23-07-1987
		ES 2003211 A	16-10-1988
		FR 2593063 A	24-07-1987
		GB 2186190 A, B	12-08-1987
		GR 870088 A	25-05-1987
		IN 166481 A	19-05-1990
		IT 1205852 B	31-03-1989
		NL 8700153 A	17-08-1987
		NZ 218927 A	06-01-1989
		PT 84146 A, B	01-02-1987
		SE 8700220 A	23-07-1987
		SG 131492 G	12-03-1993
		AT 390370 B	25-04-1990
		AT 9387 A	15-10-1989
		AU 598512 B	28-06-1990
		AU 6790387 A	23-07-1987
		BE 1000635 A	28-02-1989
		BR 8700231 A	01-12-1987
		BR 8700242 A	01-12-1987
		CA 1294557 A	21-01-1992
		CH 672250 A	15-11-1989
		DE 3701123 A	23-07-1987
		DK 35587 A	23-07-1987
		ES 2003210 A	16-10-1988
		FI 870239 A, B,	23-07-1987
		FR 2593064 A	24-07-1987
		GB 2185399 A, B	22-07-1987
		GR 870087 A	25-05-1987
		HK 19493 A	19-03-1993
		IN 168049 A	26-01-1991
		IT 1205851 B	31-03-1989
		JP 62223109 A	01-10-1987
		MX 166264 B	28-12-1992
		NL 8700152 A	17-08-1987
		NO 870253 A, B,	23-07-1987
		OA 8467 A	29-07-1988
		PH 23789 A	03-11-1989
		PT 84145 A, B	01-02-1987
		SE 8700221 A	23-07-1987
		SG 131192 G	12-03-1993
		ZA 8700051 A	31-08-1988
EP 0333301      A	20-09-1989	US 4889712 A	26-12-1989
		AT 96016 T	15-11-1993
		AU 630086 B	22-10-1992
		AU 3141589 A	21-09-1989
		BR 8901250 A	07-11-1989
		CA 1333259 A	29-11-1994
		DE 68909974 D	25-11-1993

## INTERNATIONAL SEARCH REPORT

Information on patent family members

Inten Application No

PCT/US 01/03488

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 0333301 A		DE 68909974 T	03-03-1994
		DK 132689 A	19-09-1989
		EG 19068 A	30-10-1994
		ES 2059699 T	16-11-1994
		FI 891286 A,B,	19-09-1989
		HK 206396 A	22-11-1996
		IE 64150 B	12-07-1995
		IL 89630 A	06-09-1992
		JP 2004708 A	09-01-1990
		JP 2573685 B	22-01-1997
		KR 130649 B	10-04-1998
		MX 163859 B	26-06-1992
		NO 176548 B	16-01-1995
		NZ 228351 A	27-11-1990
		OA 9240 A	30-06-1992
		PH 26841 A	05-11-1992
		PT 90020 A,B	10-11-1989
		TR 24202 A	01-07-1991
		ZA 8901979 A	28-11-1990
		ZM 1389 A	29-09-1989
US 5833954 A	10-11-1998	AU 4073097 A	06-03-1998
		BR 9711339 A	24-10-2000
		CN 1233964 A	03-11-1999
		EP 0952851 A	03-11-1999
		US 5993786 A	30-11-1999
		WO 9807448 A	26-02-1998
US 5380530 A	10-01-1995	AU 670994 B	08-08-1996
		AU 5803694 A	19-07-1994
		CA 2152813 A,C	07-07-1994
		EP 0676957 A	18-10-1995
		JP 8505140 T	04-06-1996
		WO 9414424 A	07-07-1994
JP 08301742 A	19-11-1996	NONE	
WO 9507683 A	23-03-1995	AU 7683294 A	03-04-1995
		BR 9407550 A	31-12-1996
		CA 2170489 A	23-03-1995
		CN 1130863 A	11-09-1996
		EP 0719128 A	03-07-1996
		JP 9502718 T	18-03-1997
WO 9702011 A	23-01-1997	CA 2226160 A	23-01-1997
		EP 0836470 A	22-04-1998
		JP 11509841 T	31-08-1999
		US 6193958 B	27-02-2001
US 5407661 A	18-04-1995	US 5296209 A	22-03-1994
		AT 166537 T	15-06-1998
		AU 660755 B	06-07-1995
		AU 3183393 A	22-07-1993
		CA 2086965 A	18-07-1993
		DE 69318736 D	02-07-1998
		DE 69318736 T	26-11-1998
		DK 552897 T	22-03-1999
		EP 0552897 A	28-07-1993

# INTERNATIONAL SEARCH REPORT

information on patent family members

Interr Application No

PCT/US 01/03488

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5407661 A		ES 2118183 T FI 930174 A JP 5255055 A NO 304967 B PT 101177 A,B	16-09-1998 10-11-1993 05-10-1993 15-03-1999 28-02-1994
EP 0414932 A	06-03-1991	NONE	
EP 0309414 A	29-03-1989	IT 1211746 B IT 1214424 B AU 2090488 A JP 1071445 A	03-11-1989 18-01-1990 02-03-1989 16-03-1989
US 3932603 A	13-01-1976	CA 960146 A DE 2212148 A FR 2130275 A GB 1372932 A GB 1373200 A IT 1035047 B NL 7203450 A	31-12-1974 28-09-1972 03-11-1972 06-11-1974 06-11-1974 20-10-1979 19-09-1972
US 5783172 A	21-07-1998	FR 2727979 A CA 2164799 A DE 69500114 D DE 69500114 T EP 0715844 A ES 2099646 T JP 3080871 B JP 8231362 A	14-06-1996 10-06-1996 30-01-1997 03-04-1997 12-06-1996 16-05-1997 28-08-2000 10-09-1996
US 4867989 A	19-09-1989	AU 580459 B AU 6609286 A CA 1316747 A DE 3684920 A EP 0263224 A JP 1049455 B JP 1565623 C JP 63071151 A PT 83936 A,B ZA 8701912 A	12-01-1989 17-03-1988 27-04-1993 21-05-1992 13-04-1988 24-10-1989 25-06-1990 31-03-1988 01-01-1987 31-08-1987